Two-Pass Concentration Technic Obtains Full-Flavor Grape Juice

One-stage recovery of volatiles, even at 48 percent vaporization, found inadequate. Second essence does the trick

R. P. HOMILLER, G. W. MACPHERSON PHILLIPS, R. K. ESKEW and N. H. EISENHARDT

Eastern Regional Research Laboratory, Bureau of Agricultural & Industrial Chemistry, Agricultural Research Administration, USDA, Philadelphia

OUTSIDE VIEW

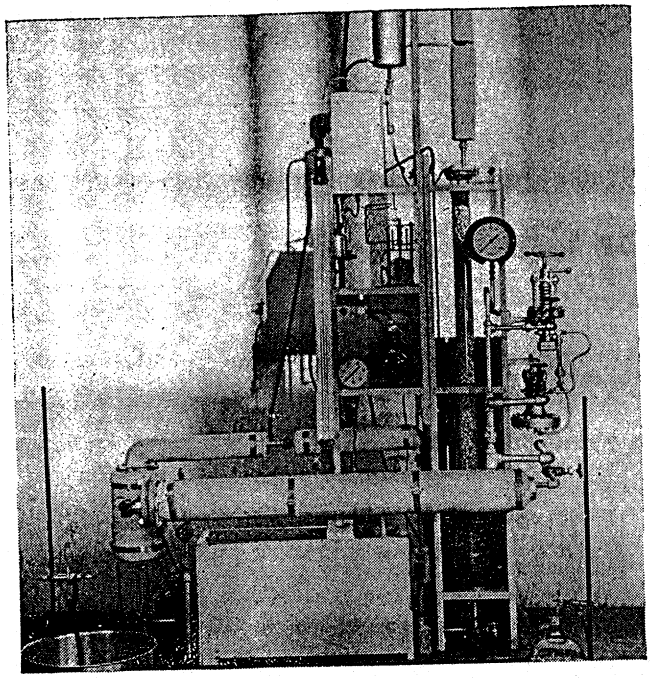

FIG. 1. UNIT used for recovery of volatile essences in two-pass method. Compact assembly is portable.

INSIDE WORKINGS

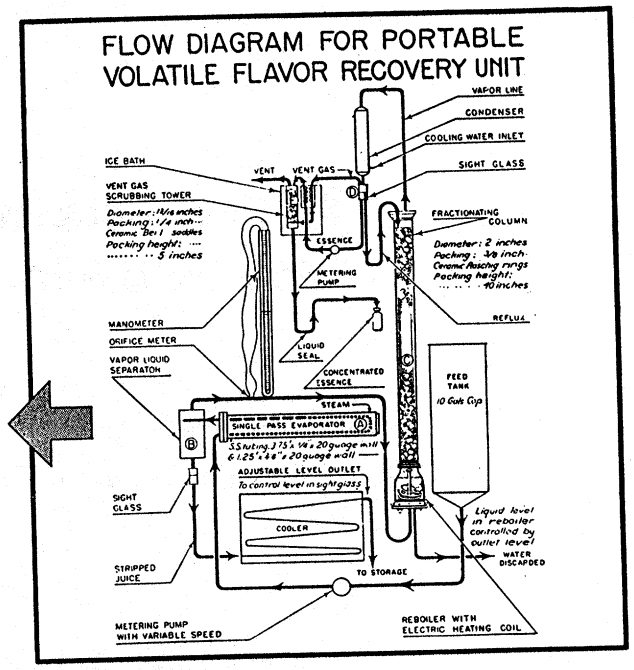

FIG. 2. HOW component parts function. Flow through apparatus is diagrammed (text explains letters).

Two-Pass Concentration Technic Obtains Full-Flavor Grape Juice

One-stage recovery of volatiles, even at 48 percent vaporization, found inadequate. Second essence does the trick

R. P. HOMILLER, G. W. MACPHERSON PHILLIPS, R. K. ESKEW and N. H. EISENHARDT

Eastern Regional Research Laboratory, Bureau of Agricultural & Industrial Chemistry, Agricultural Research Administration, USDA, Philadelphia

A Concord grape juice concentrate that has been produced by the Eastern Regional Research Laboratory reconstitutes with water into a product which has the full flavor and aroma of the original juice.

For the production of this concentrate, ERRL determined that the volatile flavors or essences of the fruit had to be recovered in two stages. These two essences are then blended with the concentrated stripped juice.

The first studies made by the Laboratory were on apple juice.* And from the interest shown in this work, the Concord grape, because of the size of the industry and the value of its products, was the next fruit selected for study.

The apparatus employed (Fig. 1) was essentially the same as that used for recovery of volatile flavors from apple juice. From 100 gal. of juice, the essence recovery process as applied to apple juice produces 10 gal. as vapor, leaving 90 gal. of "stripped juice." From the vapor are produced 1 gal. of essence and 9 gal. of water; the latter is discarded. To make a syrup concentrate, the stripped juice is evaporated under vacuum to about 15 gal., thereby removing 75 gal. of water. The concentrate and the essence are then mixed to form a full-flavor concentrate. Let us now consider the modifications in this procedure necessary to adapt it to grape juice:

Attempts were made to obtain a grape essence which would have a character and potency sufficient to enable the preparation of a full-flavor concentrate as good as the original juice. With vaporization percentages even as high as 48 percent, the addition of the essence, so obtained, to a concentrate made from the stripped juice was not quite adequate.

The resulting concentrate, when properly diluted, made a juice which had somewhat less flavor and aroma than the original juice, though its character was typical of that juice. Since the distillate obtained during the vacuum concentration of the stripped juice had a characteristic grape juice odor, our efforts were then directed toward processing this distillate into a second essence.

Because recovery of essence from this distillate is necessary in order to avoid loss, there is no point in carrying the first vaporization to any such extreme figure as 48 percent. Thus, for production of a full-flavor concentrate, the vaporization used in obtaining the first essence may well be limited to 20-25 percent of the juice.

Add Second Essence

The stripped juice is concentrated under vacuum, and the distillate obtained is passed through the essence equipment, where it is vaporized 20-25 percent to give a second essence. These

* Howard P. Milleville and Roderick K. Eskew, *Recovery and Utilization of Natural Apple Flavors,* USDA, Bur. Agr. & Ind. Chem. AIC-63, (Eastern Regional Research Laboratory) Sept. 1944, with Supplement of Apr. 1945. (Processed). Also, Howard P. Milleville and Roderick K. Eskew, "Recovery of Volatile Apple Flavors in Essence Form," *Western Canner & Packer,* Oct. 1946.

first and second essences are then added to the concentrate.

In the pilot-plant studies we determined the following significant facts:

1. A 20 to 25 percent vaporization of juice, removes, as nearly as can be determined organoleptically, all volatile flavors that can be driven off by rapid atmospheric evaporation.

2. Exposure to heat that essence-bearing vapors undergo in fractionating column and overhead system, namely, a minimum of 12 min. at approximately 212 deg. F., does not perceptibly alter flavor of reconstituted juice.

3. When producing 100-fold essence from these vapors, if vent gases are scrubbed with this essence after cooling it to 40 deg. F., loss of volatile aroma in vent gases is rather small. Term "fold" is defined as number of volumes of juice processed in making 1 volume of essence. Folds above 100 have not been investigated, but should be easily attainable with good results.

4. A continuous fractional distillation column of two plates or equivalent, with vapor feed, is all that is required to effect satisfactory production of 100-fold essence; a small reboiler at base of column is sufficient to prevent loss of flavors in waste water from bottom of rectifying column.

5. Concentration of "stripped juice" in a single effect vacuum evaporator employing a surface condenser yields, in addition to concentrate, a distillate having a characteristic Concord grape juice aroma. This is true of juices stripped at vaporizations even as high as 48 percent.

6. By processing this distillate in the essence recovery apparatus, vaporizing 20-25 percent, a "second" essence is obtained.

7. The two essences recombined with concentrated juice from which they were obtained form a full-flavor concentrate. This concentrate can be reconstituted with water to make a juice which, for all practical purposes, is equivalent to original juice.

Rapid Vaporization and Preheating Juice

To insure good recovery of the aroma in the first step, it is necessary to vaporize from 20 to 25 percent of the juice. The heating and vaporization should be rapid enough to avoid alteration of the flavor. For this purpose, the juice was pumped through a single steam-heated tube in which it was heated to its boiling point and the vaporization carried out. The retention time of the juice in the tube, using a steam pressure of approximately 15 psi. on the evaporator steam chest, was approximately 3.5 to 4.0 sec. The total time for heating, vaporizing, and cooling back to room temperature is 20 to 30 sec.

In our pilot-plant apparatus, the evaporator tube (Fig. 2, A) is fed with cold juice. The first portion of the tube acts as a preheater and the later portion as an evaporator. The point in the tube at which preheating is completed and evaporation commences will vary with the operating conditions.

This variation sometimes causes cyclical changes in the amount of vaporization and consequently pulsations in the flow through the tube. With a single tube, this can be reduced to a reasonable amount by the use of a throttling valve between the juice pump and the evaporator.

In a large commercial installation in which the rapid evaporator consists of a number of tubes in parallel, this purpose can probably best be served by a high-velocity preheater placed ahead of the evaporator and controlled to heat the juice uniformly and close to 212 deg. F.

By the vaporization, the juice becomes an intimate mixture of vapor and liquid, which must be separated. This separation is accomplished by a centrifugal separator (Fig. 2, B), from the bottom of which the unvaporized portion ("stripped juice") is removed.

A small amount of entrained juice in the vapor leaving the separator can be tolerated, for the sugar and other solids which it contains will subsequently be removed in the fractionating column. Juice so entrained is lost, however, for it will be discarded as a dilute solution in the bottoms product from the fractionating column.

Fractionation of Vapors

In the vapors, the volatile flavoring constituents are present in a concentration only 4 to 5 times that of the original juice. Since this relatively dilute product would be difficult to utilize commercially, the vapors are further concentrated in a fractionating column.

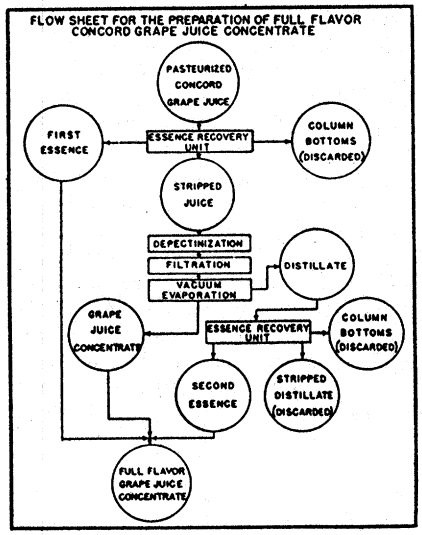

FIG. 3. SEQUENCE of operations. Note steps leading to second essence recovery.

They pass through a metering orifice into the bottom of the fractionating column (Fig. 2, C). In the pilot-plant column, a depth of 3½ ft. of porcelain Raschig rings of ⅜-in. dia. was found sufficient to effect a further 20 to 25-fold increase in concentration of volatile flavoring constituents; that is, to 100 times that of the original juice.

The vapors from the top of the column pass to a total overhead condenser. Condensed, they go to a reflux splitter or sight glass (Fig. 2, D), whence the essence is withdrawn at a rate of 1/100 that of the feed juice rate. Remainder of the distillate returns through an overflow as reflux to the distillation column (at a temperature of 170 to 190 deg. F.). A small heating coil or reboiler at the base of the column strips off any volatile flavors from the waste water before it leaves the bottom of the column.

Vent Gases Scrubbed

The juice fed to the essence equipment contains dissolved air and other gases. These pass to the fractionating column with the vapor, and must be continuously vented to the atmosphere.

To reduce the amount of volatile flavor substance carried away by these gases, they are passed up through a small scrubbing tower (Fig. 2, E), down which flows the final essence. Both the essence and the gases are cooled by ice or refrigeration before entering the scrubber, therefore the gases carry away only such small amounts of volatile materials as will saturate them at 32-40 deg. F.

If ice water instead of essence is used in the scrubber, there should be no loss of volatile flavors in the vent gases. The liquid leaving the scrubber can be returned to the column with the entering vapors, so that its essence content will be recovered. If a source of fresh odorless water is not available, or if for any reason it is undesirable to have water enter the system, the effluent from the bottom of the fractionating column can be chilled and used for this purpose.

For some juices, such as apple juice, use of water in the scrubber is an unnecessary refinement, because the vent gas from an essence-fed scrubber is odorless. For grape juice, however, the additional complication of supplying chilled water or column bottoms to the scrubber may prove justifiable.

Operation and control of the process are simple. The two constant-rate liquid pumps, one on the feed juice line and the other on the essence line, are set at the desired predetermined rates. For instance, to obtain an essence of 100-fold concentration, the essence pump is set at 1/100 of the rate of the juice pump. The amount

of vaporization, as measured by the orifice and manometer on the vapor line from the separator to the column, is controlled by regulating the steam pressure on the evaporator.

Equipment Described

Now for a brief description of the parts of the essence recovery unit used in this work (Fig. 1 and 2):

It has a working capacity of 5 gal of juice per hour at a vaporization rate of 20-25 percent. The feed tank—10-gal. capacity—is constructed of stainless steel. The metering pump, also made of stainless steel, is a gear pump with variable-speed drive. It is calibrated in terms of gph. vs. rpm. and discharge pressure.

The rapid atmospheric pressure evaporator consists of a preheating section, 3.75 ft. of ¼-in. stainless steel tubing with 20-gage wall; vaporizing section, 1.25 ft. of ⅜-in. stainless steel tubing with 20-gage wall; and steam chest, 2-in. IPS pipe provided with steam-regulating valve and condensate line.

Separator has a 4 in. dia., is 10½ in. high, and the height of inlet pipe is 7½ in. Inlet pipe is tangential to inside face of separator. The unit is all stainless steel.

Orifice meter comprises a sharp-edged ¼-in. dia. orifice connected to a water manometer calibrated in gph. of feed vaporized.

Distillation column is made up of 40 in. of ⅜-in. Raschig rings in a 2-in. dia. glass pipe. Reboiler is fitted with 500w. immersion heater.

Overhead condenser consists of a block tin coil having 0.26 sq. ft. of cooling area.

Product metering pump, capacity 16 gph., is a bellows-type positive displacement unit with external ball check valves.

Vent gas and product cooler is comprised of stainless steel tubing coils immersed in an ice water bath. Tubes have sufficient length to cool gas and liquid to 40 deg. F. or lower.

Vent gas scrubbing column has 5 in. of ¼-in. Berl saddles in a 13/16-in. dia. glass column entirely immersed in an ice water bath.

Procedure Detailed

In typical commercial manufacture of Concord grape juice, the grapes are washed and stemmed, resulting in at least partial rupture of the skin. The stemmed fruit is next transferred to a cooking tank provided with a steam jacket or coils. Here, the batch is agitated, and its temperature is raised to 130 deg. F. to develop the full characteristic Concord grape juice flavor, aroma and color. During this operation, further disintegration of the structure of the grapes occurs.

When the desired temperature has been reached, the batch is transferred, without cooling, to folded filter cloths on hydraulic press. The hot pressed juice is next pasteurized by heating to 165 to 180 deg. F., and immediately put into containers for storage. The containers are sealed and held at room temperature for 3 months to 1 year, primarily to precipitate the argol before additional processing or bottling.

The essence recovery step should obviously be applied at the processing stage at which the flavor is most fully developed. In tests made at a factory using the processing methods described above, it was found that the volatile flavors were stronger after pasteurization than at any preceding stage. Processors who age the juice to enhance its flavor and to precipitate excess argol would logically strip the essence at the completion of aging. During vacuum concentration of the stripped juice, some additional precipitation of argol occurs. With our juice, it was found that a slightly better taste resulted if this precipitate was returned to the concentrate.

Our pasteurized Concord grape juice was 20 to 25 percent vaporized to generate 100-fold first essence, as has been described. The bottoms from the fractionation column were discarded, and the stripped juice was collected (our laboratory steps are indicated in Fig. 3).

Pectin Destroyed

A pectinase enzyme was added to the stripped juice, and it was allowed to stand overnight for the complete destruction of the pectin. To destroy any excess pectinase, the juice was then heated to 140 deg. F. (with steam at atmospheric pressure) in a jacketed stainless steel kettle. It was immediately filtered on a stainless steel press using filter cloth precoated with filter-aid. Then it was passed to the evaporator for concentration.

The evaporator was of the calandria type, with a surface condenser and suitable receivers. All metal in contact with the liquid and vapor was stainless steel.

At a working vacuum of 27.5 to 28.0 in. of mercury, corresponding to batch temperatures of 99 to 109 deg. F., the juice was evaporated to a density of 65-70 deg. Brix to form a concentrate. At this density, the concentrate is self-preserving against fermentation. Commercially, if the final product is to be marketed frozen, concentration need only be carried to about 50 deg. Brix.

Second Essence Prepared

The composited distillate collected from the surface condenser during the evaporation was fed to the essence equipment. This was operated at 20 to 25 percent vaporization and 100-fold "second" essence was made. That is, 1 gal. of essence was drawn off per 100 gal. of distillate fed. Both the column bottoms and "stripped condensate" were discarded, both being essentially distilled water.

A numerical example will illustrate the quantities of the various components: If the essence equipment were operated at the 100-fold adjustment, from 100 gal. of 14 deg. Brix grape juice, there would be obtained 1 gal. of first essence, 0.6 gal. of second essence and 16.25 gal. of concentrate (58 deg. Brix). Combined, these would give 17.85 gal. of full-flavor concentrate which, on the addition of 82.15 gal. of water, would yield 100 gal. of reconstituted juice.

In commercial practice, if the essence recovery units and the juice concentrator are running continuously, it is a simple matter to combine the two streams of essence to form a mixed essence. In the instance cited above, the "fold" of the mixed essence—(i.e.) the number of gallons of juice processed in obtaining a gallon of essence—would be 100/(1 + 0.6), or 62-fold.

This essence could then be labeled "62-fold," or more simply "Mixed Essence 1.6/100", indicating that 1.6 gal. of it was made from 100 gal. of juice, and hence 1.6 gal. is the amount to be added to the concentrate made from 100 gal. of juice. If, however, the first and second essences are stored separately, they would be designated "First Essence 1/100" and "Second Essence 0.6/100", respectively, and used in these amounts.

Comparative Tests Made

Examinations of reconstituted juices in comparison with original juices (by such means as threshold dilution tests to determine the strength of the essences, and evaluation of flavor quality by a trained taste panel) indicated that the reconstituted juices were, for all practical purposes, indistinguishable from the original juices. The absence of pectin (removal of which is necessary to form a 65 deg. Brix concentrate of reasonable fluidity) did not impair the flavor of the reconstituted juice. Such absence was undetected by most of the taste panel. Storage tests on the full-flavor concentrate are being made at room temperature, in the cold, and in a frozen state.

Reconstituted grape juice prepared by the foregoing method differs slightly from commercial bottled grape juice in that the latter usually contains enough added sugar to increase the Brix from about 15 to 17 deg. Thus, to match the bottled product, sugar must be added. It can be put into the juice conveniently after the first essence has been stripped from it.